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EXAMINER

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UNITED STATES PATENT AND TRADEMARK OFFICE

BEFORE THE BOARD OF PATENT APPEALS
AND INTERFERENCES

Ex parte DOUGLAS NELSON,
THOMAS KEMMERLEY, and MICHAEL P. REMINGTON, JR.,
Appellants

Appeal 2008-3577
Application 10/652,248¹
Technology Center 1700

Decided: July 23, 2008

Before PETER F. KRATZ, CAROL A. SPIEGEL, and MARK NAGUMO,
Administrative Patent Judges.

NAGUMO, *Administrative Patent Judge.*

DECISION ON APPEAL

¹ Application 10/652,248, filed 29 August 2003, titled *Deposition of Silica Coatings on a Substrate*, referred to as the “248 Specification,” and cited as (“Spec.”). The real party in interest is listed as “Pilkington North America, which is a subsidiary of Pilkington Group Ltd, which is a subsidiary of Nippon Sheet Glass Limited, of Japan.” (Brief on Appeal filed 6 June 2007 (“Br.”), at 3.)

A. Introduction

Douglas Nelson, Thomas Kemmerley, and Michael P. Remington, Jr. (“Nelson”), appeal under 35 U.S.C. § 134(a) from the final rejection² of claims 1, 3-9, and 13-24, which are all of the pending claims. (Br. 5.) We AFFIRM.

The claimed subject matter relates to a process of depositing a silica coating on a heated glass substrate. Representative claim 1 reads as follows:

Claim 1:

A process for depositing a silica coating upon a heated glass substrate comprising the steps of:

- a) providing a heated glass substrate having a surface upon which the coating is to be deposited; and
- b) directing a precursor mixture comprising a radical scavenger, a silane, ammonia, oxygen and an inert carrier gas toward and along the surface to be coated, and reacting the mixture at or near the surface to form a silica coating on the surface of the glass substrate.

(Claims App., Br. 17; indentation added.)

Claim 16, the other independent claim, is similar, but recites that the process occurs in an on-line, float glass production process.

B. Findings of Fact (FF)

Findings of Fact throughout this Decision are supported by a preponderance of the evidence of record.

² Office action mailed 6 November 2006 (“Final Rejection”).

1. The Examiner has maintained the following rejection:³

Claims 1, 3-9, and 13-24 stand rejected under
35 U.S.C. § 103(a) in view of the combined teachings of
Soubeyrand,⁴ George,⁵ and Dick.⁶

The 248 Specification

2. The 248 Specification defines the term “silica layer” as “a coating containing primarily silicon dioxide, and possibly containing trace contaminants, for example carbon.” (Spec. 3, ¶ 7.)
3. According to the 248 Specification, it is known to form a silica-containing coating on a glass substrate by combining silane (SiH_4), oxygen, a radical scavenger gas, and a carrier gas as a precursor mixture, and then directing the precursor mixture toward and along the surface of the heated glass substrate. (Spec. 1-2, ¶ 3, citing and incorporating by reference Soubeyrand, of record in this proceeding.)

³ Examiner’s Answer mailed 20 September 2007 (“Ans.”) at 2. Two other rejections (of claims 16, 18, and 21-23 under 35 U.S.C. § 102(b) in view of Dick, and of claims 17, 19, 20, and 24 under 35 U.S.C. § 103(a) in view of Dick and Soubeyrand) have been withdrawn. (Ans. 3.)

⁴ Michel J. Soubeyrand, *CVD Method of Depositing a Silica Coating on a Heated Glass Substrate*, U.S. Patent 5,798,142 (1998).

⁵ Steven M. George and Jason W. Klaus, *Method for Forming SiO_2 by Chemical Vapor Deposition at Room Temperature*, U.S. Patent 6,818,250 B2 (16 November 2004), based on application 09/896,955, filed 29 June 2001.

⁶ Sami Dick *et al.*, *Process for the Formation of a Barrier Layer on a Surface of a Glass Object*, U.S. Patent 5,431,707 (1995). (The first inventor name is taken from the USPTO database, as S. Dick is omitted and the other inventor names are garbled on the face of the issued patent.)

4. The radical scavenger is said to allow the pyrophoric silane to be premixed with the oxygen without undergoing ignition and premature reaction at the operating temperatures. (Spec. 2, ¶ 3.)
5. A definition of the term “pyrophoric material” is “[a]ny liquid or solid that will ignite spontaneously in air at about 130F (54.4C).” *Hawley’s Condensed Chemical Dictionary*, 11th Ed. (1987).
6. The 248 Specification states that known methods of forming silica layers on substrates by chemical vapor deposition (“CVD”) processes are limited by the thickness and efficiency of the deposition process and by pre-reaction of the reactive elements, which is said to be indicated by powder formation on the substrates. (Spec. 3, ¶ 6.)
7. In one embodiment of the claimed process, silane, ammonia, a free radical scavenger such as ethylene, oxygen, and a carrier gas are combined as the precursor mixture. (Spec. 4, ¶ 8.)
8. According to the 248 Specification, “it has surprisingly and unexpectedly been found that the addition of ammonia to a known CVD process for the deposition of silica results in a silica coating with trace (less than about 1 atomic percent) to undetectable amounts of nitrogen in the silica coating.” (Spec. 7, ¶ 13.)
9. The absence of nitrogen is said to be indicated by a refractive index of about 1.45 to about 1.55. (Spec. 7, ¶ 13.)
10. The 248 Specification presents Table 1, control experiments with ammonia but without a free radical scavenger, Table 2, control experiments with ethylene but without ammonia, and Table 3, with both ethylene and

ammonia in addition to silane, oxygen, and an inert carrier gas.

(Spec. 8, ¶ 16, through 11, ¶ 18.)

11. The depositions are said to have occurred at 1170 °F (632° C) at 30 slm [standard liters per minute] and SiH₄ concentration of 1.5%.

(Spec. 8, ¶ 16.)

12. Each table reports the thickness of the resulting films, but no film compositional data is provided.

Soubeyrand

13. Soubeyrand describes a process of pyrolytically forming a silica coating on a glass substrate at elevated temperatures. (Soubeyrand 2:24-26.)

14. According to Soubeyrand, the process is especially well suited for formation of coatings on continuous float glass ribbons formed in a float glass bath enclosure. (Soubeyrand 2:26-31.)

15. Soubeyrand describes the precursor materials, which are said to comprise monosilane (SiH₄), a radical scavenger such as ethylene, oxygen, and a carrier gas, as being “directed toward and along the surface of the glass substrate” passing beneath the source of the precursor materials. (Soubeyrand 2:35-39, 55.)

16. In Soubeyrand’s words:

[t]he presence of the radical scavenger has been found to allow silane, which is pyrophoric, to be premixed with oxygen without undergoing premature ignition. Oxidation of monosilane apparently proceeds through the formation of radicals of intermediary species, and the presence of a compound acting as a radical scavenger prevents the reaction from occurring when the gas mixture is below a certain temperature threshold. [Soubeyrand 2:39-46.]

17. According to Soubeyrand, preferred materials include monosilane, ethylene, pure oxygen or atmospheric air, with nitrogen or mixtures of nitrogen and helium (to achieve a desired precursor gas density) as the carrier gas. (Soubeyrand 5:42-64.)

18. The glass temperature is said to be about 1290°F (699° C). (Soubeyrand 5:38-40.)

19. Soubeyrand does not describe the use of ammonia in the precursor gas mixture.

Dick

20. Dick describes a process in which a gaseous precursor mixture comprising silane, oxygen, ammonia, and an inert carrier gas is projected onto glass at a temperature between 300° C and the glass formation temperature, forming a barrier layer that prevents the migration of ions, especially alkali ions, out of the glass. (Dick 1:20-57.)

21. According to Dick, the carrier gas is nitrogen, argon, or a mixture of the two. (Dick 1:66-67.)

22. Deposition may be “during a final formation phase of the glass object.” (Dick 1:57-58.)

23. Dick states that in one embodiment of the process, the glass object may be coated in an “unconfined ambient atmosphere, typically free air.” (Dick 1:60-63.)

24. According to Dick, the gaseous mixture is maintained at less than 200° C, “typically less than 100° C, until its exit from the injection device.” (Dick 1:63-65.)

25. Dick teaches that an ammonia content of between 5 and 60% results, "preferably as to cost and to guarantee the formation of a dense and homogenous silicon base layer, about 50%." (Dick 1:67-2:3.)
26. Dick instructs that sodium migration data presented in the table at column 2 indicate that "the effectiveness of the chemical barrier of the deposited layer on the glass surface is substantially increased by the presence of small quantities of oxygen . . . which can be explained by the fact that the silane-oxygen reaction speed is greater than the silane-ammonia reaction speed." (Dick 2:9-15.)
27. Dick does not disclose the presence of a radical scavenger in the precursor gas mixture.

George

28. We do not find it necessary to describe George.

The Examiner's Rejection

29. The Examiner finds that the difference between the claimed process and the process disclosed by Soubeyrand is the absence, in Soubeyrand, of ammonia in the precursor gas. (Ans. 3-4.)
30. The Examiner finds that Dick teaches the use of ammonia in a precursor mixture comprising monosilane, oxygen, and nitrogen for coating a heated glass surface, to ensure a dense and homogenous silicon base layer that does not contain nitrogen. (Ans. 4.)
31. The Examiner relies on George as an alternative to Dick; thus George is essentially cumulative to Dick.

32. The Examiner concludes that it would have been obvious to use ammonia, as taught by Dick and by George in the silica coating processes in the coating processes taught by Soubeyrand for its role in forming essentially nitrogen-free homogenous silicon base layers. (Ans. 4.)

Nelson's Opposition

33. Nelson states that claims 1-9, 13, and 14⁷ stand or fall with claim 1, while claims 16-24 stand or fall with claim 16. (Br. 10.)

34. Nelson argues that the rejection of claims 1-9, 13, and 14 must be reversed in view of the unexpected results shown in the disclosure of the invention allegedly arising from the addition of both the radical scavenger (ethylene) and the ammonia. (Br. 13.)

35. In particular, Nelson urges that peak thickness is achieved with a lower percentage of NH₃ as the percentage of ethylene increases, and that the “biggest boost to coating thickness is when NH₃ is added to a gas stream containing a relatively low percentage of ethylene.” (Br. 13.)

36. Nelson urges further that the claimed invention results in the exclusion of nitrogen from the film, a result that, according to Nelson, the references do not recognize. (Br. 14.)

37. With regard to the process covered by claim 16, Nelson argues that the on-line float glass production process is not described by Dick. (Br. 14.)

⁷ Nelson does not mention claim 15 and hence does not argue for its separate patentability. Accordingly, claim 15 stands or falls with claim 1, from which it depends. 37 C.F.R. § 41.37(c)(1)(vii).

38. Nelson argues further that the production of glass receptacles, disclosed by Dick, “is inconsistent with the use of an on-line float glass production process, which produces sheets of glass. (Br. 14.)

39. Moreover, according to Nelson, the conduct of the Dick process in ambient atmosphere “differs from the on-line float glass production process claimed herein.” (Br. 15.)

C. Discussion

Claimed subject matter is not patentable if the differences from the prior art are such that the claimed invention as a whole would have been obvious to a person having ordinary skill in the art. 35 U.S.C. § 103(a). So-called secondary considerations such as long felt but unsolved needs, failure of others, etc., are to be weighed as evidence of nonobviousness if they prove instructive. *Graham v. John Deere Co. of Kansas City*, 383 U.S. 1, 17-18 (1966).

On appeal, the burden is on Nelson, as the Appellant, to demonstrate reversible error in the findings of fact or conclusions of law that underpin the Examiner’s rejections. Cf. *In re Kahn*, 441 F.3d 977, 985-86 (Fed. Cir. 2006) (“On appeal to the Board, an applicant can overcome a rejection [under § 103] by showing insufficient evidence of *prima facie* obviousness or by rebutting the *prima facie* case with evidence of secondary indicia of nonobviousness.”) (quoting *In re Rouffet*, 149 F.3d 1350, 1355 (Fed. Cir. 1998)); *In re Mayne*, 104 F.3d 1339, 1344 (Fed. Cir. 1997) (“Even were it obvious to a practitioner of the art [that the results were unexpected], applicants have the burden to provide the PTO with evidence showing such is the case.”).

Nelson's argument that claims 16-24 are not obvious over the references because Dick's teaching of coating receptacles is inconsistent with a float glass process is not persuasive. Nelson has not directed our attention to any evidence in the record that suggests that those skilled in the art are so unsophisticated that they would be directed away from combination with Soubeyrand, which does teach coating a continuous float glass ribbon during its formation within a float glass bath enclosure by Dick's reference to coating "objects." A continuous float glass ribbon is, after all, an object. Moreover, Dick teaches that the glass object may be coated at temperatures ranging from 300° C up to the temperature of formation of the glass. (Dick 1:54-58; FF 21.)

Nelson's argument that Dick's teaching of coating in an ambient atmosphere differs from the process covered by claim 16 is without merit. Dick's reference is to "one aspect of the invention [in which] the projection of the gaseous mixture on the hot surface is effected in an unconfined ambient atmosphere, typically free air." (Dick 1: 60-63.) On its face, the teaching is not limited to "free air." Nor has Nelson directed our attention to any credible evidence that the specific embodiments described by Dick at column 2, lines 39-47, necessarily require projection onto hot objects maintained in free air.

Nelson has not argued against—and has therefore conceded in this proceeding—that the Examiner has established a *prima facie* case of obviousness of claims 1-9, and 13-15 in view of the combined teachings of Soubeyrand, George, and Dick.

Nelson's argument that the exclusion of nitrogen from the coating film is unexpected fails for several reasons. First, the nitrogen content of the coating film is not recited in any of the claims, except for claim 6, which Nelson has indicated it does not argue separately from claim 1. (FF 33; Br. 10.) Second, the specification lacks sufficient evidence, including comparative evidence, supporting this proposition. Evidence of non-obviousness must be objective factual evidence, not merely argument or conclusory statements of the applicant. *In re DeBlauwe*, 736 F.2d 699, 705 (Fed. Cir. 1984) (“Mere argument or conclusory statements in the specification does not suffice.”) DeBlauwe did not present any experimental data showing that prior heat shrinkable articles split; due to the absence of tests comparing appellants' heat shrinkable articles with those of the closest prior art, the court in *DeBlauwe* concluded that appellants' assertions of unexpected results constituted mere argument); *see also In re Geisler*, 116 F.3d 1465, 1470 (Fed. Cir. 1997) (Geisler's proof of unexpected results failed for lack of evidence.) This failing applies to all the claims, including claim 6, which specifies that the level of nitrogen in the film be less than “about 1 atomic percent.” Absent comparative results, there is no basis to ascertain whether one of ordinary skill in the relevant arts would have considered the alleged levels of nitrogen obtained by the presently claimed processes to be unexpected. Second, Nelson has failed to challenge the Examiner's finding (FF 30) that Dick teaches such an exclusion of nitrogen from the coating film.

Nelson's argument that the peak thickness is obtained with less ammonia as the amount of ethylene increases is an unexpected result that overcomes the *prima facie* case of obviousness also fails because the data

offered are not commensurate in scope with the claimed processes. *In re Grasselli*, 713 F.2d 731, 743 (Fed. Cir. 1983) (“It is well settled that objective evidence or non-obviousness must be commensurate in scope with the claims which the evidence is offered to support.”) (internal quote and citation omitted.) Nelson’s claimed processes are not limited by relative amounts of any components, nor by the temperatures of the glass substrate or of the reactants. Both the prior art (Soubeyrand (FF 16) and Dick (FF 26)) and the 248 Specification (FF 3, 4, 6) indicate that the relative kinetics of various reactions play important roles in the process conducted and the product formed. When several competitive reactions are occurring simultaneously, complex behavior is not necessarily unexpected. Although Nelson has observed the effect of relative concentrations of ammonia and ethylene on silica layer thickness, Nelson has not explained why that effect would have been unexpected to a person having ordinary skill in the art. Nor has Nelson explained why the argued beneficial results obtained at a single substrate temperature, under a single delivery rate, over a limited range of compositions, would have been understood to be fairly representative of the entire range of exclusive protection Nelson seeks.

D. Summary

In view of the record and the foregoing considerations, it is:

ORDERED that the rejection of claims 1-9, 13, 14, and 16-24 under 35 U.S.C. § 103(a) in view of the combined teachings of Soubeyrand, Dick, and George is AFFIRMED;

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FURTHER ORDERED that no time period for taking any subsequent action in connection with this appeal may be extended under 37 C.F.R. § 1.136(a).

AFFIRMED

qsg

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